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Monitoring of pilot plants in 2012 - 2014

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LIVESTOCK RESEARCH
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# Summary

From 2009 to 2011 the agricultural, economic and environmental effects of the production and use of mineral concentrates, produced from animal slurry, were studied. Part of the study was the monitoring of the 8 participating full-scale (pilot) plants to assess the chemical composition of the half products and the end products of the process. In 2012 - 2014 the monitoring programme was continued with 9 pilot plants. In this programme additional data from the pilot plants were collected on the chemical composition of the raw slurry and the end products. Samples from these process streams were analysed on dry matter, volatile solids and the main nutrients N, P and K. The raw material of 8 pilot plants was pig slurry only. One plant used effluent from a biogas plant mixed with pig slurry as a feedstock.

The production process included 3 steps: (1) solid/liquid separation of the raw slurry using a belt press, a screw press or a drum filter, (2) clearing of the liquid fraction by means of air flotation or micro filtration and (3) concentration of the minerals by means of reverse osmosis.

The average concentrations of the main valuable components N and K in the mineral concentrate produced in 2012-2014 were 6.9 and 6.7 g/kg resp., which is a fraction lower than in the period 2009/2011. The proportion of ammonia (TAN) in total-N was 90%. The average concentration of P in the concentrate was 0.19 g/kg or 0.44 g/kg phosphate ($P_2O_5$).

Quality standards for mineral concentrates were established to guarantee the product has fertilizing characteristics that are comparable with chemical fertilizer, is harmless to the environment and is distinguishable from other animal manures. The next monitoring programme will be focused on checking the mineral concentrates, produced by the participating pilot plants, against the quality standards.

# Samenvatting

Tussen 2009 en 2011 heeft het landbouwbedrijfsleven, het ministerie van Economische Zaken en het ministerie van Infrastructuur en Milieu in de pilot mineralenconcentraten onderzoek laten uitvoeren naar de landbouwkundige, economische en milieukundige effecten van de productie en het gebruik van mineralenconcentraat ter vervanging van kunstmest. Het onderzoek betrof onder andere het monitoren van 8 pilotinstallaties met het doel de chemische samenstelling van de ingaande mest en de eindproducten vast te stellen. Daarnaast werd een literatuurstudie gedaan naar de natuurlijke afbreekbaarheid van hulpstoffen (polyacryl-amiden) en werden enkele manieren verkend om het mineralenconcentraat te upgraden. Van 2012 tot 2014 werd de monitoring vervolgd met 9 pilotinstallaties waarbij aanvullende gegevens over de chemische samenstelling van de processtromen werden verzameld. Monsters van deze processtromen werden geanalyseerd op droge stof, organische stof en de belangrijkste nutriënten N, P en K. De ingaande mest van 8 pilotinstallaties bestond uit varkensdrijfmest. Eén installatie gebruikte een mix van varkensdrijfmest en digestaat uit een biogasinstallatie die met onder meer co-producten werd gevoed.
Het productieproces bevatte de volgende 3 processtappen: (1) scheiding van de ruwe mest in een dikke en een dunne fractie d.m.v. een zeefbandpers, een vijzelpers of een trommelfilter, (2) zuiveren van de dunne fractie door middel van flotatie of microfiltratie en (3) concentratie van de minerale vloeistof door middel van omgekeerde osmose.

De gemiddelde concentraties van de belangrijkste waardevolle componenten N en K in het mineralen concentraat dat in de periode 2012-2014 werd geproduceerd bedroeg resp. 6.9 en 6.7 g/kg, een fractie lager dan in de periode 2009-2011. Het aandeel minerale stikstof ($NH_4$-N) in de totale stikstof bedroeg gemiddeld 90%. De gemiddelde concentratie P in het concentraat bedroeg 0.19 g/kg of 0.44 g/kg fosfaat.

Er werden kwaliteitseisen voor mineralenconcentraat vastgesteld om er zeker van te zijn dat het product bemestingseigenschappen bezit die vergelijkbaar zijn met die van kunstmest, niet schadelijk is voor het milieu en te onderscheiden is van andere mestsoorten. In de volgende monitoringsperiode zullen de mineralenconcentraten, geproduceerd door de deelnemers aan de pilot, worden getoetst aan de kwaliteitseisen.

# 1 Introduction

Manure processing in the Netherlands is considered as one of the options to relieve the pressure on the manure market, others being feed modification and export of manure. One possible route is the production of a mineral concentrate that can be applied as a replacement for chemical fertilizer.
From 2009 to 2011 the agricultural, environmental and economic effects of the production and use of mineral concentrate was studied, contributing to the strive of environmentally safe distribution of manure and closure of mineral cycles. If mineral concentrates are recognized (by the European Commission) as chemical fertilizers, they can be applied additionally to the application standard for nitrogen from animal manure.
Within the framework of the pilot mineral concentrates from 2009 to 2011 a monitoring programme was executed to assess the chemical composition of mineral concentrates and get knowledge of the fertilizer value and the environmental effects of the production and application as substitutes of chemical fertilizers. The results show that a mineral concentrate can be considered as a liquid fertilizer with ammonia and potassium as the main valuable components (Hoeksma et al., 2011; Hoeksma & de Buisonjé, 2012). The mineral concentrate also contained small quantities of volatile solids, phosphorus, dissolved salt, heavy metals and micro-contaminants. Mineral concentrates if responsibly used have no negative effects on the environment (Ehlert & Hoeksma, 2011). The C/N ratio of mineral concentrates was 7.5 to 8.5, values at which denitrification processes in the soil can progress with reasonable speed. Part of the carbon in the mineral concentrates was present as easily degradable volatile fatty acids.
To avoid loss of nitrogen due to denitrification the carbon content of the mineral concentrate should be as low as possible. Quality requirements of mineral concentrates should be related to minimal N loss after application to the soil.
Field trials showed that the N-efficiency of mineral concentrate on arable land and grass land was 84% and 58% respectively as compared with granulate chemical fertilizer (calcium-ammonium-nitrate). The N-efficiency was comparable with liquid ammonium-nitrate both on arable land and on grass land (van Geel et al., 2011a; Middelkoop & Holshof, 2011). Additional field trials showed that with mineral concentrate N-efficiencies equal to calcium-ammonium-nitrate can be achieved if it is correctly applied at the right time (van Geel et al., 2011b).

In 2012 - 2014 the monitoring programme was continued with 9 pilot plants. In this programme additional data from the pilot plants were collected on the chemical composition of the raw slurry and the end products. Samples from these process streams were analysed on dry matter, volatile solids, the main nutrients N, P and K, acidity (pH) and electrical conductivity (EC). The raw material of 8 pilot plants was pig slurry only. One plant used effluent from a biogas plant mixed with pig slurry as a feedstock. Results of the monitoring are presented in this report. The chemical composition of mineral concentrates as produced in the period covered by this report was compared with quality requirements keeping environmental protection and differentiation from other liquid manure fractions in mind.

# 2 Materials and methods

## 2.1 Production process

The general concept of the production process of a mineral concentrate from livestock manure as studied in this project is shown in Figure 2.1. Raw pig slurry is separated into a solid fraction and a liquid fraction after adding a coagulant and a flocculant. Suspended solids and colloid particles are removed from the liquid fraction by air flotation. The permeate from air flotation is filtered and subsequently concentrated by reverse osmosis, leaving a mineral concentrate and an effluent (permeate).

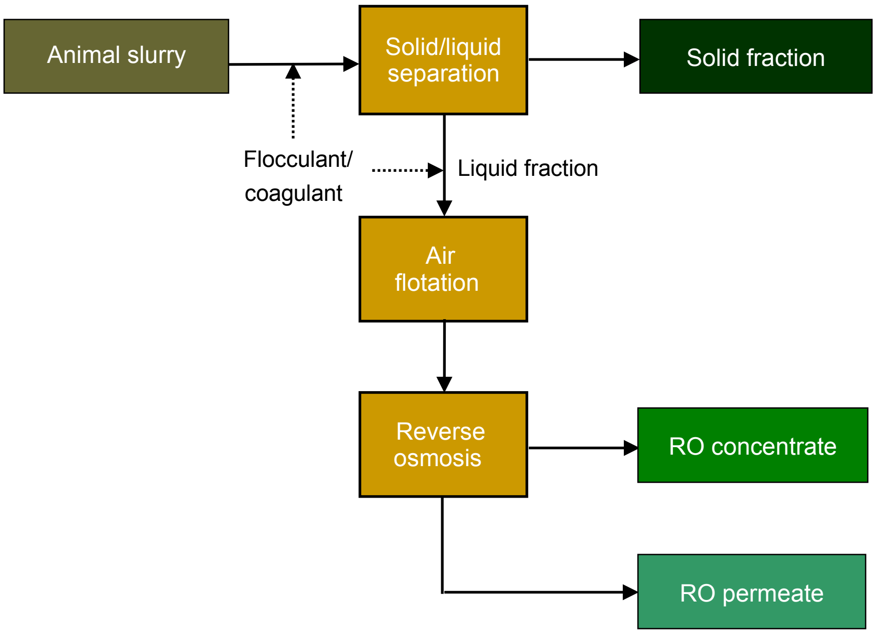


***Figure 2.1*** *Production process of mineral concentrate using reverse osmosis*

The principle of reversed osmosis is based on the ability of RO-membranes to let water pass and block salt ions. Water is pressed through the membrane. The pressure needed for the process to take place depends on the conductivity (EC value) of the feed. During the process the conductivity of the concentrate increases and an increasing pressure is needed to get an almost clean permeate. The maximal degree of concentration is limited by the osmotic pressure of the feed and the resulting driving force. The treatment plants participating in the project apply RO-membranes which were originally designed for desalination of sea water.

## 2.2 Pilot plants

From the start in 2009 a total of 13 plants (coded A-H, K-N and P) participated in the pilot. Due to technical or economic reasons 4 plants quit producing mineral concentrates. In the period 2012-1014, covered by this report, 9 pilot plants were monitored, identified by their original codes. The main characteristics of the plants are given in Table 1.

Table 1
*Characteristics of pilot plants that were monitored in 2012 – 2014*

| Plant code | Monitoring from | Feed stock | Capacity (ton/year) | Solid/liquid separation | Polishing liquid fraction | Additives used |
|---|---|---|---|---|---|---|
| B | 2012 | Pig slurry | 80.000 | Belt press | Air flotation | Coagulant*<br>Flocculant** |
| C | 2012 | Pig slurry | 25.000 | Belt press | Air flotation | Flocculant |
| D | 2012 | Pig slurry | 10.000 | Screw press | Air flotation | Flocculant |
| F | 2012 | Pig slurry | 80.000 | Belt press | Air flotation | Flocculant |
| G | 2013 | Pig slurry | 30.000 | Belt press | Air flotation | Coagulant<br>Flocculant |
| K | 2013 | Pig slurry<br>Digestate*** | 36.500 | Drum filter | Micro filtration | Flocculant |
| L | 2013 | Pig slurry | 25.000 | Belt press | Air flotation | Coagulant<br>Flocculant |
| M | 2013 | Pig slurry | 16.000 | Belt press | Air flotation | Flocculant |
| P | 2014 | Pig slurry | 25.000 | Belt press | Air flotation | Flocculant |

* *Coagulant = iron (III) sulphate ($Fe_2(SO_4)_3$)*
** *Flocculant = polyacrylamide*
*** *Digestate = effluent from a biogas production plant fed with pig slurry and co-products (maize, starch rest, coffee grounds, sludge, glycerine) on a 50/50 (w/w) basis.*

## 2.3 Monitoring programme

During the monitoring period the performance of the pilot plants was assessed by measuring the chemical composition of the raw slurry and the three end products solid fraction, RO-concentrate and RO-permeate. The amounts of N, P and K in these process streams were also quantified. Table 2 shows which parameters were measured.

Table 2
*Measured parameters in raw slurry and end products of the pilot plants*

| |
|---|
| Total solids (TS), volatile solids (VS) |
| Total Nitrogen (Total-N), Total Ammonia Nitrogen (TAN), Phosphorus (P), Potassium (K) |
| Acidity (pH) |
| Electrical conductivity (EC) |

N, P and K are valuable fertilizing compounds and quality indicators of the end products, as well as VS. Electrical conductivity and pH are important process control parameters.

## 2.4 Sampling

The process streams were sampled three times per year. Four pilot plants (B, C, D and F) were sampled during the entire monitoring period of three years. Four plants (G, K, L and M) were sampled during two years and one plant (P) only one year because of later start-up. From one plant (N) no representative samples could be collected due to technical problems and revision work.
For sampling liquid process streams (raw slurry, RO-concentrate and RO-permeate) we used existing valves in the tubing of the plant from which 1 litre samples were taken. The solid fraction was sampled directly after solid/liquid separation by collecting 1 litre sample from the transport conveyer belt. Working this way 'fresh' samples were collected.

## 2.5 Chemical analysis

Raw slurry and solid fraction were analysed according to the methods as prescribed by the Dutch manure law (Accreditation programme animal manure; composition AP05). Table 3 gives an overview of the Dutch standards that were followed for each parameter.

Table 3
*Dutch standards for chemical analysis of the process streams.*

| Parameter | Dutch standard | Description |
|---|---|---|
| Total solids + ash[1] | NEN 7432: 1998 nl | Animal manure and manure products – determination of total solids and volatile solids content – gravimetric method |
| Total N (Kjeldahl) | NEN 6641: 1983 nl | Sludge – determination of the sum of the mineral nitrogen (ammonia) and organic nitrogen content according to Kjeldahl after mineralization using selenium |
| TAN | NEN 7438: 1998 nl | Animal manure and manure products – determination of ammonia nitrogen content – titrimetric method |
| TAN | NEN-ISO 7150-1:2002 en | Water – determination of ammonia<br>Part 1: hand spectrophotometric method |
| P[2] | NEN 6662 | |
| K | NEN 6442: 1997 nl | Water – flame photometric determination of the potassium content |
| pH | NEN 6411: 2006 | Water and sludge – determination of acidity (pH) |
| Electrical conductivity (EC) | NEN ISO 7888: 1994 en | Water – determination of electrical conductivity |

[1] *Volatile solids is calculated as difference between total solids and ash*
[2] *Phosphate ($P_2O_5$) is calculated from P as P*2.29*

All chemical analyses were carried out by the laboratory of environmental research of Wageningen-UR.

## 2.6 Data processing

The average value and standard deviation of the parameters measured in raw slurry and end products overall and of individual pilot plants are presented. The composition of the end products of individual plants was compared for total solids, volatile solids, total-N, TAN, P and K. Regression and variance analysis were executed with Genstat 13$^{nd}$ edition.

# 3 Results

## 3.1 Composition of raw slurry

Table 4 shows the average composition of the raw slurry (feed stock) of individual pilot plants in 2012 – 2014.

Table 4
*Average composition of raw slurry fed to pilot plants in 2012-2014 (in g/kg)*

| Plant | B | C | D | F | G | K | L | M | P |
|---|---|---|---|---|---|---|---|---|---|
| TS | 65.2 | 59.7 | 50.3 | 70.4 | 45.1 | 96.8 | 44.9 | 92.4 | 36.9 |
| VS | 43.8 | 41.2 | 32.6 | 45.3 | 27.0 | 71.2 | 29.5 | 67.2 | 23.6 |
| Total- N | 6.08 | 5.94 | 4.25 | 5.95 | 4.09 | 7.24 | 3.59 | 7.85 | 2.38 |
| TAN | 3.81 | 3.90 | 2.42 | 3.76 | 2.30 | 2.92 | 2.13 | 4.26 | 1.14 |
| P | 1.55 | 1.28 | 1.31 | 1.44 | 0.87 | 1.75 | 1.03 | 1.60 | 0.70 |
| K | 4.19 | 3.68 | 2.36 | 3.89 | 2.68 | 4.20 | 2.42 | 4.63 | 1.99 |
| pH | 7.75 | 7.63 | 7.75 | 7.82 | 7.16 | 7.72 | 7.61 | 7.83 | 7.38 |
| EC (mS/cm) | 29.1 | 28.3 | 20.0 | 29.4 | 24.4 | 25.8 | 21.5 | 31.7 | 15.0 |

Table 4 shows clear differences in raw slurry composition between the pilot plants. The inputs of plants B, C, F, L and M was mainly slurry from fattening pigs, the inputs of plants D, G and P mainly sow slurry. Notice the relatively low concentrations of the measured components in the raw pig slurry used as input of plant L which are nearly similar to the concentrations in sow slurry (input of plant G). The feedstock of plant K was pig slurry 50/50 mixed with the effluent (digestate) from the biogas production plant (at the same location); the biogas plant was fed with a mixture of pig slurry and organic co-products (see Table 1).

## 3.2 Composition of solid fraction

Table 5 shows the average composition of the solid fraction after mechanical solid/liquid separation of each pilot plant in 2012-2014. Different separation technologies were used (see Table 1). All plants used polyacrylamide as a flocculant to improve the separation efficiency. Plants B, G and L used iron III sulphate ($Fe_2(SO_4)_3$) as an additive. Figure 3.1 shows the composition of the solid fraction relative to raw slurry.

Table 5
*Average composition of solid fraction of pilot plants in 2012-2014 (in g/kg)*

| Plant | B | C | D | F | G | K* | L | M | P |
|---|---|---|---|---|---|---|---|---|---|
| TS | 309 | 261 | 230 | 306 | 277 | 875 | 303 | 316 | 379 |
| VS | 231 | 204 | 156 | 237 | 195 | 651 | 223 | 255 | 278 |
| Total- N | 13.7 | 12.7 | 10.4 | 13.0 | 12.2 | 22.9 | 11.8 | 12.4 | 13.9 |
| TAN | 5.13 | 5.39 | 3.56 | 5.09 | 3.73 | 2.18 | 3.53 | 4.21 | 2.87 |
| P | 7.21 | 6.38 | 7.08 | 7.42 | 6.07 | 14.2 | 7.11 | 6.26 | 8.64 |
| K | 3.85 | 3.46 | 2.50 | 3.97 | 3.03 | 14.7 | 2.63 | 3.50 | 3.32 |

* *Solid fraction was post treated in a SHS (super-heated steam) dryer using heat from a heat and power unit which is fueled with biogas.*

The TS content of the solid fraction varies from 230 to 379 g/kg which corresponds with what could be expected from the separation technologies used. No correlation was found between the TS content of the raw slurry and the TS content of the solid fraction nor between the contents of other components. So the raw slurry has not significantly affected the composition of the solid fraction. In 2009 – 2010 a significant effect of the raw slurry was found for TAN and K (Hoeksma et al, 2011).

The effectivity of the solid/liquid separation was different between the pilot plants. This is illustrated by Figure 3.1 in which the composition of the solid fractions are compared with the raw slurries. The figure shows that TS, VS and P are concentrated in the solid fraction, most effectively at plant P: lowest concentrations in the raw slurry and highest in the solid fraction. Plants G and L performed relatively well, whereas the performance of plant M was relatively poor. In general the 'new' plants performed better than the 'old' plants. The performance of the latter was comparable to the performance in 2009-2010. In this comparison plant K is left out of consideration because the solid fraction was post treated.

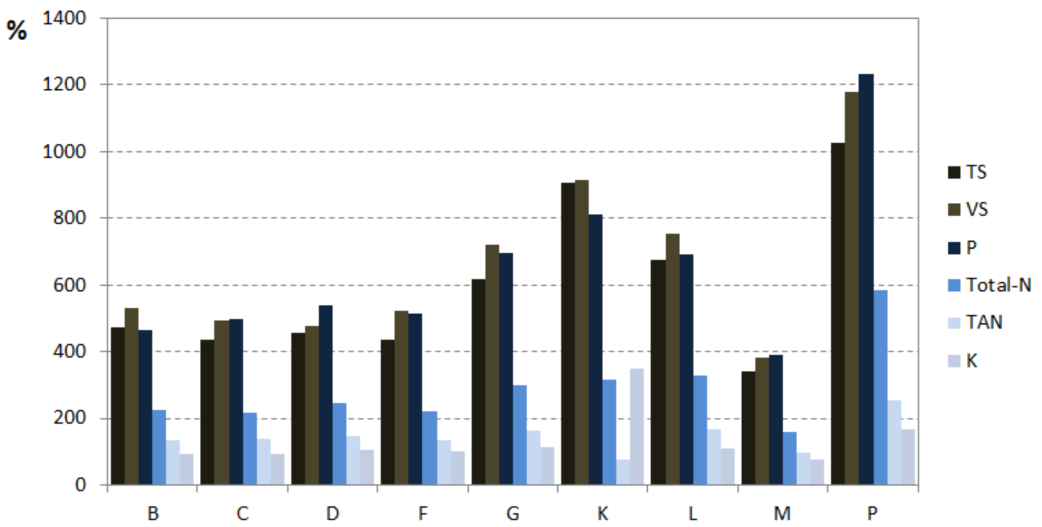


**Figure 3.1** *Content of TS, VS, total-N, TAN, P and K in solid fraction, relative to raw slurry. Content in raw slurry is 100%.*

## 3.3 Composition of RO concentrate

Table 6 shows the average composition of the RO-concentrate produced by individual pilot plants in 2012 - 2014. Figure 3.2 shows the composition of the RO-concentrates relative to raw slurry.

Table 6
*Average composition of RO-concentrate of individual pilot plants in 2012-2014 (in g/kg)*

| Plant | B | C | D | F | G | K | L | M | P | Overall Average |
|---|---|---|---|---|---|---|---|---|---|---|
| TS | 37.7 | 34.5 | 29.8 | 33.6 | 30.6 | 36.6 | 34.6 | 33.9 | 31.4 | **33.6** |
| VS | 11.1 | 15.4 | 11.4 | 14.4 | 8.76 | 14.2 | 11.2 | 14.8 | 10.6 | **12.4** |
| Total- N | 7.43 | 7.51 | 6.41 | 8.13 | 6.83 | 6.98 | 5.65 | 8.09 | 5.20 | **6.91** |
| TAN | 7.02 | 6.43 | 5.75 | 7.02 | 6.22 | 6.59 | 5.31 | 7.11 | 4.50 | **6.22** |
| P | 0.04 | 0.26 | 0.13 | 0.24 | 0.16 | 0.54 | 0.04 | 0.23 | 0.05 | **0.19** |
| K | 7.93 | 6.94 | 6.38 | 7.18 | 6.36 | 6.66 | 5.60 | 6.75 | 6.64 | **6.72** |
| pH | 7.88 | 8.00 | 7.96 | 8.03 | 7.86 | 7.74 | 7.59 | 8.07 | 7.73 | **7.87** |
| EC | 63.6 | 54.7 | 52.7 | 58.7 | 56.4 | 57.7 | 52.9 | 58.6 | 50.7 | **56.2** |

Table 6 shows differences in composition of the RO-concentrate between the pilot plants. Plant P produced a concentrate with low concentrations of the nutrients total-N, TAN and K compared with the other plants. The highest nutrient concentrations are found in the concentrates of plants B, F and M. The concentrate of plant K shows a remarkably high P content, which raises questions about the effectivity of the pre-treatment, consisting in mechanical solid/liquid separation with a drum filter and micro-filtration, for removal of P. The overall average composition of the RO-concentrate produced in 2012-2014 is similar to the average RO-concentrate as produced in 2009. From 2009 to 2011 the RO-concentrate showed higher and increasing concentrations of total-N, TAN and K.

The RO-concentrate of plant P shows the highest nutrient concentrations compared with the raw slurry, as is illustrated by Figure 3.2. The concentrates of the other pilot plants show slightly higher nutrient concentrations as compared with the raw slurry that was used as feedstock. The average concentration of total-N is a factor 1.5 and of TAN and K a factor 2.0 higher in the RO-concentrate compared with raw slurry. The concentrations in the RO-concentrate of VS and P are approximately 30% resp. 10% of the concentrations in raw slurry.

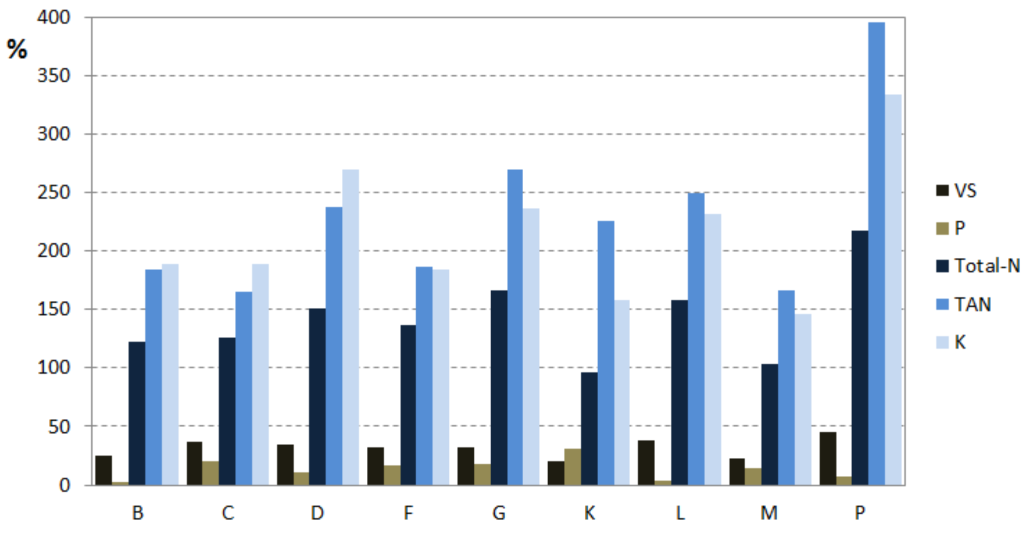


***Figure 3.2*** *Content of VS, total-N, TAN, P and K in RO-concentrates, relative to raw slurry. Content in raw slurry is 100%.*

### 3.3.1 Quality requirements

Results obtained from the monitoring have led to quality requirements for RO-concentrates that guarantee an effective agronomic working of the product, harmless to the environment and distinguishable from other animal manures. The quality requirements are:

1. TAN/Total-N ≥ 90%
2. Total-N/$P_2O_5$ ≥ 15
3. EC ≥ 50 mS/cm.

The TAN/Total-N criterion is laid down to ensure that nitrogen in RO-concentrates is mostly present as mineral nitrogen. The Total-N/$P_2O_5$ and EC criterions are introduced to be able to distinguish RO-concentrates from other animal slurries. The EC value of a slurry can quickly be checked by means of a handheld instrument.
The average Total-N content of RO-concentrates lies within the range of the Total-N content of animal slurries as produced in Dutch livestock farming. The average $P_2O_5$ content of RO-concentrates is lower than of slurries. A low phosphate concentration differentiates RO-concentrates from most livestock slurries but after slurry dilution comparable $P_2O_5$ concentrations can be obtained.
The Total-N/$P_2O_5$ ratio of RO-concentrates is much higher than of other slurries and therefore is a distinguishing criterion.
The portion of TAN in Total-N is considered as an important feature to characterize RO-concentrates; the higher the TAN/Total-N ratio the more RO-concentrates look like chemical fertilizers.

Table 7 shows the average concentrations of Total-N, TAN and $P_2O_5$, as well as TAN/Total-N and Total-N/$P_2O_5$ ratios and EC values of RO-concentrates of the pilot plants as measured in 2012-2014.

Table 7
*Average concentrations (in g/kg) of Total-N, TAN and $P_2O_5$, as well as TAN/Total-N and Total-N/$P_2O_5$ ratios and EC values of RO-concentrates in 2012-2014.*

| Plant | B | C | D | F | G | K | L | M | P |
|---|---|---|---|---|---|---|---|---|---|
| Total- N | 7.43 | 7.51 | 6.41 | 8.13 | 6.83 | 6.98 | 5.65 | 8.09 | 5.20 |
| TAN | 7.02 | 6.43 | 5.75 | 7.02 | 6.22 | 6.59 | 5.31 | 7.11 | 4.50 |
| $P_2O_5$ | 0.09 | 0.60 | 0.30 | 0.55 | 0.37 | 1.24 | 0.09 | 0.53 | 0.11 |
| TAN/Total-N | 0.94 | *0.86* | 0.90 | *0.86* | 0.91 | 0.94 | 0.94 | *0.88* | *0.87* |
| Total-N/$P_2O_5$ | 83 | *13* | 21 | 15 | 18 | *5.6* | 63 | 15 | 47 |
| EC | 64 | 55 | 53 | 59 | 56 | 58 | 53 | 59 | 51 |

Table 7 shows that the RO-concentrates of 4 pilot plants (B, D, G and L) in the monitoring period 2012-2014 on average answered to all quality requirements. All RO-concentrates met the EC-50 requirement. Most of the RO-concentrates that do not meet the TAN/Total-N 90% requirement (C, F and M) show a high $P_2O_5$ content (>0.50 g/kg). The concentrates involved also show a (too) low Total-N/$P_2O_5$ ratio. A low TAN/Total-N ratio and high $P_2O_5$ content of RO-concentrate are related to insufficient pre-treatment of the raw slurry with regard to removal of volatile solids; N and P in animal slurry are part of organic structures to a certain extent.
Some liquid manure fractions and intermediate products from manure processing, such as effluents from ultra-filtration and air flotation, can meet the standards for TAN/Total-N and Total-N/$P_2O_5$ ratio's. None of these fractions and products can meet the EC-50 standard (Hoeksma et al., 2011).

Although there are variations in the composition of RO-concentrate produced by an individual pilot plant all plants except plant K have shown that they are able to produce a concentrate that meets all requirements. The position of plant K in this respect is exceptional because the feedstock from which RO-concentrate is produced is partly digestate from a biogas plant, while all other plants use pig slurry. There are indications that separation of digestate and subsequent polishing of the liquid digestate fraction is more difficult than of livestock slurry only (Hjorth et al., 2010).
Some producers will have to make an extra effort to meet the quality requirements for RO-concentrates and realize a product with constant quality.

## 3.4 Composition of RO permeate

Table 8 shows the average composition of RO-permeate produced by individual pilot plants in 2012 - 2014.

Table 8

*Average composition of RO-permeate of individual pilot plants in 2012-2014 (in g/kg)*

| Plant | B | C | D | F | G | K | L | M | P |
|---|---|---|---|---|---|---|---|---|---|
| TS | 0,11 | 3,90 | 1,07 | 0,38 | 0,20 | 0,24 | 0,08 | 0,36 | 0,02 |
| VS | 0,01 | 1,37 | 0,14 | 0,09 | 0,07 | 0,07 | 0,02 | 0,09 | 0,01 |
| Total- N | 0,04 | 1,17 | 0,45 | 0,28 | 0,08 | 0,06 | 0,01 | 0,12 | 0,02 |
| TAN | 0,04 | 1,07 | 0,44 | 0,23 | 0,08 | 0,06 | 0,01 | 0,12 | 0,02 |
| P | 0,00 | 0,05 | 0,00 | 0,00 | 0,00 | 0,00 | 0,00 | 0,00 | 0,00 |
| K | 0,03 | 0,88 | 0,33 | 0,08 | 0,03 | 0,03 | 0,01 | 0,06 | 0,01 |
| pH | 7,41 | 8,55 | 8,14 | 9,52 | 8,23 | 8,20 | 5,24 | 7,18 | 8,13 |
| EC | 0,11 | 3,90 | 1,07 | 0,38 | 0,20 | 0,24 | 0,08 | 0,36 | 0,02 |

The RO-permeate is the end product (‘water’) that is not used as a fertilizer because it contains very low concentrations of nutrients, as is shown in Table 8. Ideally the permeate is discharged to the surface water (free of charge) or reused in the farm as cleaning water. However, disposal to surface water is not always allowed because the permeates do not meet the legal discharge standards for total-N, TAN and P (Hoeksma et al, 2011).

Theoretically dissolved particles do not pass a reverse osmosis membrane, only gases (such as ammonia ($NH_3$)) pass through the membrane. However, poor construction or damage of the membrane may open the possibility that ions and dissolved particles pass the membrane. The monitoring of the pilot plants learned that in all day practice small amounts of ions end up in the RO-permeate. The quality of the permeate can be improved by using ion exchange as a post treatment step, which is practiced by plant B and plant L.

## 3.5 Post treatment

### 3.5.1 Solid fraction

*Biogas production*

Solid fractions typically contain a high quantity of organic matter and therefore is a feedstock with biogas potential. The solid fractions of most of the pilot plants used to find their way to biogas plants in the nearby or not so nearby area. However, the cost of transport is substantial, adding up to € 5 to the treatment cost per ton of raw slurry. The cost of disposal of the solid fraction is increasing due to new legislation that forces livestock farmers to process (export or incinerate) part of their manure surplus based on N and P. Today more and more producers are looking for alternative ways to use the solid fraction.

*Composting/pasteurization*

Composting is a biological aerobic process. During the composting process organic material is converted by microorganisms. Composting is an exo-energetic process; temperatures may rise up to 70$^{o}$C or more without external heating, resulting in a dry and stable pasteurized product. Composting of solid manure is operated in windrows in open air, in tunnels with forced aeration or in a closed (rotating) drum. In the latter case it is a more controlled operation, although the residence time in the rotating drum (e.g. 24 hours) is too short to obtain a stable end product. If the composting process is performed well, the end product is exportable.

*Drying/pasteurization*

If cheap (free) thermal energy is available, such as heat from a combined heat and power unit (CHP), it is attractive to use this energy for drying and pasteurization of the solid fraction. One of the pilot plants (plant K) is operating a highly efficient super-heated steam (SHS) dryer which is using heat from its own heat and power unit.

Today, the main purpose of heating solid fractions is to produce a pasteurized exportable product. Often heating is followed by granulation to make handling more easy and to enlarge the market.

### 3.5.2 RO concentrate

RO-concentrates are characterized by relatively low concentrations of fertilizing components being ammonia ($NH_4^+$) and potassium (K) mainly. The value of the concentrates as such is relatively low and transport cost is high. The market for RO-concentrates is small even if the user would pay the same price as chemical fertilizer relative to the fertilizer value. Upgrading, meaning higher concentrations of N and K and volume reduction, would increase the value of the concentrates and broaden the market. During the period of the pilot a number of technologies were superficially tested (within the framework of another project) to upgrade RO-concentrates, the one more cost-effective than the other.

*Evaporation using ventilation air from the pig house*

Water from the RO-concentrate is evaporated by applying the concentrate as recirculation liquid in an air scrubber for a pig house after acidifying it to ensure that the ammonia stripped from the ventilation air is fixed in the concentrate. The electrical conductivity (EC) is used as process control parameter. The end product is characterized by smaller volume and substantially higher ammonia-nitrogen content as compared with the raw RO-concentrate. Test results indicate that 40% volume reduction and an increase of the N content with a factor >10 is possible. The process is characterized by a high sulphuric acid demand, which is a serious drawback of this application.

As a version of this concept the RO-concentrate is recirculated in a scrubber-like evaporation unit which is placed between the ventilator outlet and the air scrubber. This version leaves two end products. The ventilation air will take up moisture and is stripping ammonia, leaving a concentrate with reduced volume and low ammonia and high potassium content. The ventilation air is treated in the scrubber leaving diluted acid with a high ammonia content. Tests are to be continued.

*Filtration by Trans Membrane Chemo Sorption (TCMS)*
In this filtration process the RO-concentrate is heated and next treated with lye to shift the $NH_4^+/NH_3$ towards the gaseous $NH_3$. Subsequently the concentrate is filtered over the TMCS-membrane. Gaseous ammonia passes the TMCS-membrane and is transferred to a strong acid solution (sulphuric, nitric or phosphorus acid) at the other side of the membrane. The driving force of the process is difference in concentration between the liquids on both sides of the membrane. The process leaves two end products: an effluent with low ammonia content and a nitrogen-concentrate.
From the test results it was concluded that an end product with 50 g N/kg is possible (Hoeksma & de Buisonjé, 2012). The efficiency of the TMCS process should be further improved, which can be found in optimization of the process temperature (energy demand), pH of the treated liquid (lye demand) and the liquid flows over the TMCS-membrane.

Evaporation and filtration of the RO-concentrate means that products with different properties are created. Data on the agricultural efficiency of RO-concentrates that were found during the pilot do not necessarily apply to these products.

### 3.5.3 RO permeate

*Ion exchange*
At plants B and L the permeate from the RO is post treated by ion exchange. The purpose of ion exchange is to ensure that the permeate meets the requirements for ammonium nitrogen for discharge on surface water (< 5 - 15 mg N/litre), thus avoiding the cost of discharge into the sewer. The permeate from the RO still contains some nitrogen, existing for more than 90% of ammonium ions ($NH_4^+$), typically > 20 mg N/litre, requiring further polishing before being discharged onto surface water.
Ion exchanging selectively removes ammonium from the RO permeate. The ion exchange unit consists of polyester tanks, filled with porous polymer resin beads (polystyrene with sulphonate groups) that holds cations like $Na^+$ or $H^+$ that can be exchanged for other cations, such as ammonium ions ($NH_4^+$). The resin has a higher affinity for the ammonium cation than for the other cations. The ammonium ions are thus adsorbed by the resin while the exchanged $Na^+$ or $H^+$ cations are discharged with the permeate. The efficiency of an ion exchanger is > 90% in general, but the resin has to be regenerated periodically in order to remain effective.

# 4 Discussion

RO-concentrates as produced by the pilot plants are characterized as N-K fertilizers with relatively low concentrations of valuable components and correspondingly low fertilizer value. On average the quality of the RO-concentrates during the first three years (2009-2011) of the monitoring slightly improved but showed a small decay over the last three years. An increasing number of producers recognize the necessity to improve the quality of RO-concentrates to ensure the sales as a substitute for chemical fertilizer. They face the challenge of finding a cost-effective way to reduce the volume of the RO-concentrate. Evaporation, using ventilation air from the pig house in combination with an air scrubber, and trans membrane chemo sorption are considered as options, although the high energy and acid consumptions of both options are a serious drawback. Likewise, in order to make solid fractions export worthy, some producers have taken initiatives to test alternative technologies such as heating, drying and composting for pasteurization of the solid fraction.

The production of RO-concentrates supports the ambition to reuse of minerals from animal manure as much as possible and to reduce the nitrogen surplus at national level under one condition: RO-concentrate has to be recognized by the European Commission as a mineral fertilizer that can be applied additionally to the legal application standard for N from animal manure.

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